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Title: Extraction of Nitroaromatic and Nitroamine Explosive Compounds and Picric Acid from Soil Samples [SW-846 8330A & 8330B]

Approvals (Signature/Date)

[redacted]

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1.0 Scope and Application

1.1 This standard operating procedure (SOP) describes the extraction of nitroaromatic and nitroamine explosive residues from soil samples. This procedure is based on SW-846 8330A and 8330B, but can also be performed on samples for analysis by method SW-846 8321A.

1.2 This procedure does not describe the analysis of the extracts. For those details, see the following SOPs:

1.2.1 DV-LC-0002 "*Analysis of Nitroaromatic and Nitroamine Explosive Compounds by HPLC"*

1.2.2 DV-LC-0010 "*Analysis of Nitroaromatic and Nitroamine Explosives Compounds by APCI/LC/MS"*

1.2.3 DV-LC-0025 *"Analysis of Picric Acid by LC/MS/MS"*

1.3 Application of 8330A versus 8330B

1.3.1 This procedure is for extraction by either Method 8330A or 8330B. The most important differences in the two source methods are the more rigorous sample collection and preparation measures in 8330B, which are designed to produce more representative results. The more rigorous 8330B process is specifically intended to complement the incremental field sampling process described in Appendix A of method 8330B. If incremental or equivalent systematic sampling processes are not employed in the field, then the extra laboratory homogenization and subsampling effort 8330B requires may add little or no improvement in the overall precision of results.

1.3.2 A larger sample size is used for 8330B (10 g) than is used for 8330A (2 g). A larger sieve size is used for 8330B (10 mesh) than is used for 8330A (30 mesh)

2.0 Summary of Method

2.1 Solid samples are air dried to a constant weight and sieved. Soil agglomerates are broken with a mortar and pestle, sieve shaker, or mechanical grinder. For samples requiring the more rigorous homogenization techniques found in method 8330B, the analyst employs a ring and puck grinder. The samples are extracted with a 0.1% acetic acid in acetonitrile mixture on a shaker table.

3.0 Definitions

3.1 Definition of terms used in this SOP may be found in the Glossary section of the TestAmerica Denver Quality Assurance Manual (QAM) or SOP DV-QA-003P, *Quality Control Program.*

3.2 Explosives: As used in this SOP, the term "explosives" refers specifically to the analytes listed in Table 1. These include compounds that can be readily detonated with heat, shock, or ignition, such as nitroglycerin, RDX, and TNT. It also includes production by-products and degradation products of true explosives.

3.3 TALS: TestAmerica Laboratory Information Management System

3.4 ISM: Incremental Sampling Methodology - This is a requirement of method 8330B and describes the technique used to take a 10g aliquot from a sample in at least 30 increments.

3.5 Extraction Holding Time: The elapsed time expressed in days from the date of sample collection to the date the extraction starts. The holding time is tracked in the laboratory LIMS system, and is the primary basis of prioritizing work.

3.6 Preparation Batch: A group of up to 20 samples that are of the same matrix and are processed together in the same extraction event using the same procedure and lots of reagents and standards

3.7 Grinding Batch: A grinding batch is up to 20 samples processed through the same grinding procedure. When using the ring and puck mill, the grinding batch is opened with a grinding LCS and a grinding blank and must be closed after 20 samples or after 3 days, whichever is sooner, due to the expiration of the grinding LCS**.**

3.8 Method Comments: The Method Comments are used to communicate to the bench level chemists special requirements and instructions from the client.

3.9 Quality Assurance Summary (QAS): Certain clients may require extensive specific project instructions or program QC, which are too lengthy to fit conveniently in the Method Comments field in LIMS. In these situations, laboratory Project Managers describe the special requirements in a written QAS to address these requirements. QASs are posted on a public drive for easy accessibility by all lab employees. Normally, QASs are introduced to analysts in an initial project kick-off meeting to be sure that the requirements are understood.

3.10 Aliquot: A part that is a definite fraction of a whole; as in "take an aliquot of a sample for testing or analysis." In the context of this SOP, "aliquot" is also used as a verb, meaning to take all or part of a sample for preparation, extraction, and/or analysis.

4.0 Interferences

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interferences, under the conditions of the analysis, by running method blanks.

4.2 Contamination by carryover can occur when a low-concentration sample is extracted immediately following a high-concentration sample.

4.3 Samples from an ammunition plant or depot usually contain analytes that were deposited via water and leaching and therefore are more uniformly dispersed. Therefore, as per SW-846 8330B Section 11.1.4.2, ring and puck is not necessary.

4.4 Samples from firing ranges and impact zones can contain particles of explosives at a variety of sizes, shapes, and compositions. Therefore the entire sample must be processed through a ring and puck prior to removal of the subsample for analysis. Samples collected at the firing point can contain nitrocellulose fibers. These fibers present a special problem in the grinding step. In order to get the fibers to release the target analytes they must be very finely ground. For these samples only the ring and puck should be used. The client needs to be consulted when selecting a grinding mechanism.

4.5 Tetryl decomposes rapidly with exposure to heat as well as methanol/water solution. All samples expected to contain tetryl should not be exposed to temperatures above room temperature.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 Specific Safety Concerns or Requirements

5.2.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated will be removed and discarded; non-disposable gloves must be cleaned immediately. When tightening caps on 40 mL glass vials, cut resistant gloves must be worn.

WARNING: Soil samples with explosive concentrations greater than 2% cannot be accepted by the laboratory unless they have moisture content of 25% or greater. Under no circumstances shall a soil sample with an explosive concentration greater than 10% be accepted by the laboratory.

5.2.1.1 If a sample is expected to have an explosive concentration ≥2% (but less than 10%), the EH&S Coordinator and Group Leader shall be notified before any work is performed. Additional safety precautions may be implemented as required due to high concentrations of explosives.

5.2.1.2 Soil samples with high concentrations (between 2 and 10%) of explosives should not be ground using a mortar and pestle. Visual observation of a soil samples is important prior to grinding samples. Any samples containing metal fragments, powders, waxy appearing pieces, or other suspicious material should be brought to the attention of the Group Leader and the EH&S Coordinator before proceeding with the procedure. Bypassing the grinding step and proceeding to solvent dilution is an alternative for samples that are determined to be unsafe to grind.

5.2.2 Anyone working in the grinding room needs to be enrolled in the Hearing Conservation Program. See SOP DV-HS-0010 for details. Personnel operating the grinding equipment are required to wear ear plugs when the equipment is turned on. When standing next to the Humbolt mechanical grinder described in Section 6.1.11 during operation, the decibel levels are above 80 decibels, therefore anyone operating the grinder must be enrolled in the Hearing Conservation Program and wear hearing protection. While the grinder is running, the decibel levels in the room are below 80 decibels, therefore personnel not enrolled in the Hearing Conservation Program can be in the room. Hearing protection is always available to every analyst and they are encouraged to use it.

5.2.3 Operations involving handling samples outside of sealed containers are conducted in ventilation hoods to avoid exposure to dust. Dust masks are available for use, but are optional.

5.2.4 Operations involving the grinding of radioactive samples can be particularly hazardous due to the increased potential for exposure from airborne dust. If a sample is labeled as a “CAT 1”, “CAT 2”, “CAT 3” or “CAT 4” sample, and requires grinding thru the ring and puck, contact the RSO immediately.

5.3 Primary Materials Used

The following is a list of materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not contain all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.

A complete list of materials used in the method can be found in the reagent and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

MATERIAL [1]	HAZARDS	EXPOSURE LIMIT [2]	SIGNS AND SYMPTOMS OF EXPOSURE
ACETONITRILE	Flammable Poison	40 PPM – TWA	Early symptoms may include nose and throat irritation, flushing of the face, and chest tightness. Prolonged exposure to high levels of vapors may cause formation of

MATERIAL [1]	HAZARDS	EXPOSURE LIMIT [2]	SIGNS AND SYMPTOMS OF EXPOSURE
			cyanide anions in the body.
METHANOL	Flammable Poison Irritant	200 PPM - TWA	A slight irritant to the mucous membranes. Toxic effects are exerted upon the nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness, and dizziness. Methyl alcohol is a defatting agent and may cause the skin to be become dry and cracked. Skin absorption can occur, symptoms may parallel inhalation exposure. Irritant to the eyes.
ACETIC ACID, GLACIAL	Corrosive Poison Flammable Liquid and Vapor	10 PPM - TWA	Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur. Can cause serious damage to skin, including redness, pain, and burns. Contact with eyes may cause severe damage followed by loss of sight.
(1) ALWAYS ADD ACID TO WATER TO PREVENT VIOLENT REACTIONS. (2) EXPOSURE LIMIT REFERS TO THE OSHA REGULATORY EXPOSURE LIMIT.			

6.0 Equipment and Supplies

6.1 Equipment

All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

6.1.1 Balance, capable of measuring ± 0.01 g. Calibration checked per SOP DV-QA-0014

6.1.2 Orbital shaker table, capable of maintaining 150 rpm for 18 hours.

6.1.3 Pipettor with disposable 1.0 mL tips, accurate to ± 2%, calibration checked daily in accordance with SOP DV-QA-0008.

6.1.4 Bottle-top pipettor, able to dispense 8 to 20mL, accurate to ± 2%, calibration checked daily in accordance with SOP DV-QA-0008. If the pipettor does not have a digital display, then the calibration check should be performed whenever the pipette is adjusted.

6.1.5 Ring and Puck - for the grinding of soils per method 8330B

The grinding bowl and puck are cleaned after each use by washing with soap and water with a plastic brush, rinsing with hot tap water, rinsing with DI water, and then rinsing with a 10% acetonitrile solution in acetone. A final wipe down of the bowl and puck while still wet with solvent is done with a Kimwipe (TNT in particular is reported to be prone to adhering to steel surface). In addition, sand blanks are used to monitor potential carry-over for each batch of samples (see Section 9.10.1 for details).

6.1.6 Trays – “baker’s rack” type of stack for the air drying of soils per method 8330B

6.1.7 Sieves, 10 and 30 mesh – Sieves are cleaned after each use by washing with soap and water and a green plastic brillo pad, (be careful not to damage the mesh), rinsing with hot tap water, rinsing with DI water. Prior to use, the sieves are rinsed with 10% acetonitrile in acetone and wiped with a Kim Wipe. Sieves are allowed to dry in a hood prior to use.

6.1.8 Receiver pans and lids – Receiver pans are cleaned after each use by washing with soap and water, rinsing with hot tap water, rinsing with DI water. Prior to use, the receiver pans are rinsed with a 10% acetonitrile in acetone and wiped dry with a Kim Wipe.

6.1.9 Sieve shaker – used to facilitate the sieving of large sample volumes.

6.1.10 Mortar and pestle – cleaned after each use by washing with soap and water, rinsing with hot tap water, and then rinsing with DI water. Prior to use, the mortars and pestles are rinsed with 10% acetonitrile in acetone and wiped with a Kim Wipe and allowed to dry in a hood prior to use.

6.1.11 Mechanical Grinder – Humbolt Manufacturing Part Number H-4199. Used in place of a mortar and pestle to quickly reduce cakes of dry soil. The grinder reduces soil agglomerates and sieves the soil through a 10 mesh sieve. The mechanical grinder is used to break up soil agglomerates, but it is not an alternative to Ring and Puck. The mechanical grinder is cleaned after each sample by removing the hopper. The hopper is washed with soap and water, rinsed with tap water, rinsed with DI water, and then rinsed with 90:10 Acetone:Acetonitrile. The Hopper is then wiped dried with a laboratory tissue. The hammers and body of the grinder are cleaned after each sample by rinsing with DI water and wiping dry with a laboratory tissue.

6.2 Supplies

6.2.1 Glass vials, various sizes.

6.2.1.1 Amber glass, 40 mL, with Teflon-lined screw caps for the sonication of soil samples.

6.2.1.2 Amber glass, 8.0 mL, with Teflon-lined screw caps, for the storage of final extracts.

6.2.2 Aluminum foil and aluminum dishes.

6.2.3 Parchment paper

6.2.4 0.2-μm PTFE syringe filters and disposable syringes.

6.2.5 Wooden spatulas – used to lay samples out to dry.

6.2.6 Plastic square-ended disposable spoons – used to aliquot samples.

6.3 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1 Ottawa Sand – baked at 400°C for at least 4 hours.

7.2 Acetonitrile, CH_3CN - HPLC grade (ACN). Each lot is tested per CA-Q-S-001 DV-1.

7.3 Soil Extraction Solvent – approximately 0.1% acetic acid in acetonitrile – Open a new 4-liter bottle of acetonitrile and add 4 mL of acetic acid, then cap and mix. This reagent is given a 1 year expiration date.

7.4 Standards

Please reference SOP DV-OP-0020 for information regarding the surrogate and spike standards used in this procedure.

7.5 Grinding LCS Bulk Material

A standard is purchased in a matrix of -20/+70 Sieved Soil that contains the compounds at the concentrations listed in Table 2. This standard comes packaged in 500 g containers. This standard is stored in a refrigerator at 0°C to 6°C and is given a 1 year expiration date. After grinding the ground LCS is stored refrigerated and has a three day expiration date.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Soil samples to be extracted by method 8330A for analysis by method 8330A should be collected in eight-ounce wide mouth jars with Teflon-lined caps. When sampling for DoD projects that must comply with DoD QSM, version 3, 4 or 5 requirements for drying and sieving the entire contents of a soil sample container, a separate container should be used to collect a soil sample for this analysis.

8.2 For soil samples to be extracted by method 8330B for analysis by either method 8330B or method 8321A, it is not uncommon to receive samples of 1 kg or more. Samples may be shipped in wide mouth jars or clean plastic bags.

8.3 Sample extracts must be stored refrigerated in amber glass containers at ≤ 6 °C and not frozen.

8.4 Soil and sediment samples should be air dried at ambient temperature until dry enough to sieve. See Section 10.3 for details. Once the sample is air dried, the sample can be stored at room temperature.

8.5 All soil and sediment samples must be extracted within 14 days of collection and analyzed within 40 days after extraction begins.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Soils	Glass/ plastic	4 grams (8330A)/up to 1 Kg (8330B)	Cool ≤ 6°C	14 Days	SW846 8330A/B

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs. This procedure meets all criteria for DoD QSM 5.0 unless otherwise stated. Any deviation or exceptions from QSM 5.0 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 11.0 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument. See QC Policy DV-QA-003P for further details.

Grinding Batches – A grinding batch is up to 20 samples processed through the same grinding procedure. When using the ring and puck mill, the grinding batch is opened with a grinding LCS and a grinding blank and must be closed after 20 samples or after 3 days, whichever is sooner, due to the expiration of the grinding LCS.

9.4 Method Blank (MB)

A method blank (MB) must be prepared and analyzed with each batch of samples. The MB consists of Ottawa sand with surrogates added. The MB is created at the time of extraction after the samples have been dried, sieved, and ground and is then carried through all extraction and analysis steps. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false-positive data.

9.5 Laboratory Control Sample / Laboratory Control Sample Duplicate (LCS/LCSD)

One LCS must be analyzed with each batch of samples. The LCS must contain specified analytes of interest and must be carried through the entire analytical procedure. The LCS is prepared by spiking the analytes of interest into Ottawa sand. The LCS is created at the time of sample extraction after the samples have been dried, sieved, and ground. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

NOTE: DoD requires the MS/MSD to be assigned by the client. When there is no assigned MS/MSD or there is not enough sample volume provided a LCSD is not required unless requested by the client.

9.6 Matrix Spike Sample (MS) and Matrix Spike Duplicate (MSD)

A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. The soil matrix spikes are created at the time of extraction. Spikes and surrogate compounds are added after the sample has been dried, sieved, and ground. One MS/MSD pair must be processed for each preparation batch. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process.

If insufficient sample volume is available for MS/MSD, a LCSD must be performed and an NCM must be written.

NOTE: DoD requires the MS/MSD to be assigned by the client. When there is no assigned MS/MSD or there is not enough sample volume provided a LCSD is not required unless requested by the client.

9.7 Surrogate Spikes

Every calibration standard, field sample, and QC sample (i.e. method blank, LCS, LCSD, DU, TRL, MS, and MSD) is spiked with surrogate compounds.

9.8 Sample Duplicate (DU)

A duplicate sample is required after ring and puck grinding is performed. A duplicate sample is also required for method 8330B, even if grinding is not performed. A sample duplicate is a second aliquot of one of the samples in the batch. Field blanks cannot be used for duplicate testing. The results for duplicates are reported separately, and cannot be averaged when reporting results. Sample duplicate results are used to evaluate the precision of the method. As such, results should be greater than or equal to the RL for a valid statistical comparison.

9.9 Sample Triplicates (TRL)

A triplicate sample is required after ring and puck is performed. A triplicate sample is also required for method 8330B, even if grinding is not performed. The lab will determine the %RSD as defined below. Results for the %RSD as well as the individual replicate results will be reported to the client. The method suggests that the %RSD for the subsampling error is acceptable if it is <10%. For DoD QSM 5.0, the %RSD is acceptable if it is <20% for results above the LOQ.

The percent relative standard deviation (%RSD) is calculated as follows:

$$\%RSD = \frac{s}{\bar{C}} \times 100\%$$

Where s is the standard deviation of the average concentration and is calculated as follows:

$$s = \sqrt{\frac{\sum_{i=1}^{n} (C_i - \overline{C})^2}{n-1}}$$

In the event that the laboratory is requested to perform the evaluation of field replicate precision, three field replicates designated by the client will be processed through the entire homogenization and extraction steps. The %RSD for these replicates will be calculated as indicated above and reported to the client.

9.10 Grinding Blank (GB)

9.10.1 Ring and Puck Grinding Blanks

Before each sample is processed through the ring and puck mill, the ring and puck will be cleaned per Section 6.1.5. Then approximately 200 g of Ottawa Sand will be ground. This ground sand will be saved and labeled with the sample ID of the next sample ground with the suffix “blank”. After a batch of samples has been processed through the ring and puck, a composite will be generated using sub-aliquots from all blanks ground before the samples. This is done by placing approximately 1 tablespoon of material from each of the individual sample blanks in a clean re-sealable plastic bag. The bag is then sealed and the material is mixed and homogenized by shaking and kneading the bag. A 10g aliquot is then removed from the bag and labeled as the batch grinding blank. This composite is extracted and analyzed in the same manner as the field samples.

Corrective Action: If the composite grinding blank results are greater than the acceptance limits, then the individual grinding blanks will be extracted and analyzed to determine when the contamination occurred and exactly which samples were affected. Samples associated with a contaminated grinding blank producing positive results for the same contaminant, must be reprocessed and reanalyzed. If un-ground sample is not available, then the potential carry-over between samples must be described in a non-conformance memo and discussed in the final report case narrative.

9.11 Grinding LCS (LCSSRM)

One Grinding LCS must be ground and analyzed with each batch of samples that are processed through the ring and puck. The Grinding LCS must contain specified analytes of interest and must be carried through the entire analytical procedure. The Grinding LCS is prepared by grinding a 500 g aliquot of the Grinding LCS Bulk Material described in Section 7.5 without having air-dried the material before hand. The Grinding LCS must be ground using the same grinding apparatus (ring and puck) as the samples were ground. The Grinding LCS is used to monitor the effects of the grinding process on the analytes of interest. On-going monitoring of the LCS results

provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

Corrective Action: If the Grinding LCS fails the acceptance criteria, samples associated with the Grinding LCS must be reprocessed and reanalyzed. If un-ground sample is not available, then the results of the grinding LCS must be described in a non-conformance memo and discussed in the final report case narrative.

10.0 Procedure

NOTE: Rotate sieves and any applicable equipment; do **not** use specific sieves or equipment for the MB and LCS/LCSD.

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP # DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Dry the Samples – Refer to the Flowchart in Appendix 1 and the batching instructions in Appendix 3

10.3.1 Check the Method Comments to see if the samples are for a project with the Department of Defense (DoD), If yes, then the entire contents of the sample container must be dried. Check TALS to make sure the client sent more than one container if additional tests are being requested. If additional tests are logged and the client only sent one container, the Project Manager should be notified.

10.3.2 If the sample is logged for method 8330B, or for an ISM method, then the entire contents of the sample container must be dried. Check each sample to make sure the client sent more than one container if additional tests are being requested. If additional tests are logged and the client only sent one container, the Project Manager should be notified

10.3.3 If the samples do not fall under the descriptions given in Section 10.3.1 or Section 10.3.2 then only a portion of the sample container needs to be dried. In these cases, lay out at least 20 g to dry.

10.3.4 Depending on the sample size, the samples are laid out in aluminum pans, or on large trays lined with aluminum foil to dry. Some clients may request metals analysis on the dried samples. In those cases, samples are laid out on parchment paper.

10.3.5 Spread the samples out in a thin layer to facilitate drying. Use a disposable wooden spatula to break up any clumps and agglomerates.

10.3.6 The tray or pan that the sample is laid out into is labeled with the sample ID. A second analyst checks to make sure that the labels on the tray or pan match the labels on the client sample container to ensure samples are not accidently mixed up. This check is documented in TALS.

10.3.7 Place the samples in a hood or well ventilated area at room temperature. Document in TALS the date and time the samples were laid out to dry. If the samples are very wet, a fan can be used to help facilitate the drying process, but care should be taken so that the air flow is not strong enough to cause cross-contamination between samples. An electronic temperature recording device records the temperature of the room and the data is downloaded weekly.

10.3.8 When the samples appear to be dry enough that they can be sieved without caking, subsample approximately 15 grams into an appropriate weighing vessel and record the exact weight, the date, and the time (see Appendix 5). Set this 15 gram aliquot (still in the weighing vessel) next to the rest of the drying sample. Take care to use an appropriate weighing vessel for the analytical methods requested, as the aliquot removed in this step will still be included in the volume used for ISM (i.e. Do not use an aluminum weigh boat for samples requiring metals analysis).

10.3.9 After 2 hours, reweigh the aliquot in the same weighing vessel and record the exact weight, the date, and the time. If the weight of the sample is within 10% of the previous weight, proceed to Section 10.4.

NOTE: The procedure for verifying that samples have been dried to a constant weight, as described in the previous two sections, is included for DOD compliance. For non-DOD samples only, it is acceptable for an experienced analyst to verify by sight that the samples are dry in the interest of meeting turnaround times or holding times. In the case that the constant weight determination is not performed, an NCM must be generated for deviation from this SOP. However, the constant weight verification procedure must be performed and documented for all DOD samples.

10.4 Sieve the Samples - Refer to the Flowchart in Appendix 2.

10.4.1 If the client requirements specify a particular sieve size, those instructions take precedence.

10.4.2 If the sample is logged for prep method “8330_P_2g” then a 30 mesh sieve should be used.

10.4.3 If the sample is logged for prep method “8330_Sonc_10g” then a 10 mesh sieve should be used.

10.4.4 Some clients will request metals analyses to be performed on the sieved sample. In those cases, a stainless steel sieve should be used. Brass sieves should be avoided.

10.4.5 Clean the sieves prior to use following the instructions in Section 6.1.7.

10.4.6 Some samples may require the use of a mortar and pestle or a mechanical grinder to break up dried clumps. Refer to Sections 6.1.10 and 6.1.11 on how to clean and rinse the mortar and pestles and the mechanical grinder before use.

10.4.7 Sieve the entire dried sample through the appropriate sized sieve. Care must be exercised not to eliminate soil agglomerates during this step. The soil can be broken into small pieces with a gloved hand or another instrument (a wooden spatula for example). If a gloved hand is used, care should be taken to change out gloves in between samples so not to cross- contaminate samples.

10.4.8 Remove large rocks, vegetation, and twigs that do not pass through the sieve. Mosses and other types of fine vegetation should be physically shredded while sieving to release trapped soil and residues. The only materials that should be eliminated by sieving are rocks and vegetation. All soil must be broken up to pass through the sieve.

10.4.9 Place any soil that does not pass through the sieve into a clean mortar. Break up soil agglomerates using the pestle. Or as an alternative use the mechanical grinder. Be sure to break up all soil so that it can pass through the sieve. Only extraneous material such as rocks and vegetation should be removed with the sieve. Describe all extraneous material that did not pass through the sieve in an NCM.

NOTE: Some clients may request the portion of the sample that did not pass through the sieve to be saved and weighed. Some clients may request the weight of the entire sample to be documented as well. Check Method Comments before discarding any sample material.

10.4.10 Collect all of the material that passes through the sieve on a clean piece of foil or parchment paper.

10.4.11 An automatic sieve shaker can be used to help facilitate the sieving of samples. A receiver pan is placed under a sieve and the sample is added to the sieve with 1 or 2 small grinding stones. Then a lid or another receiver pan for a second sample is placed on top. The stack is then clamped inside the sieve shaker for no more than 30 minutes. Inspect the samples to ensure that only extraneous material such as rocks and vegetation should be removed with the sieve. If needed use a mortar and pestle to break up soil agglomerates. Describe all extraneous material that did not pass through the sieve in an NCM.

10.5 Grind the Samples - Refer to the Flowchart in Appendix 2.

10.5.1 If the samples are not logged with a pre-prep method of "ISM_DD_SI_PM_SS" skip this section and proceed to Section 10.6.

10.5.2 Ring and Puck Grinding *Samples logged for "ISM_DD_SI_PM_SS"*

10.5.2.1 See Section 6.1.5 on how to clean the ring and puck dish.

10.5.2.2 If the sample is logged for ring and puck grinding, a grinding blank per Section 9.10.1 consisting of baked Ottawa sand will be processed through the ring and puck dish before each sample. These individual blanks will be composited into one grinding blank for the associated samples and will be analyzed in addition to the normal extraction blank.

NOTE: When preparing the grinding blanks, it is not necessary to do five 60-second grinds. One 60-second grind of the Ottawa sand is sufficient.

10.5.2.3 After a grinding blank has been processed through a ring and puck dish, that blank is labeled as the blank associated to the next sample processed through that same dish. Do not clean the ring and puck dish after the blank.

10.5.2.4 Prepare a grinding LCS per Section 9.11 with every batch. The grinding LCS will be analyzed in addition to the normal extraction LCS.

NOTE: A grinding batch will consist of no more than 20 samples that have been ground within three days of each other. The grinding batch is opened with a grinding LCS and a grinding blank and must be closed after 20 samples or after 3 days, whichever is sooner**.** A grinding batch must have one Grinding LCS, and at least one Grinding Blank. If more than one Grinding Blank is prepared, it must be very clear on the benchsheet which individual sample blanks were used to build each Grinding Blank.

10.5.2.5 In a hood, transfer the sample into a clean ring and puck dish. Do not overfill the dish (approximately 300 g of sample can fit in one dish). If needed, grind the sample in 300 g or smaller increments and recombine after all sample has been ground. The entire sample must be ground. Place the dish securely in the holder and close the door on the machine. Grind the sample in five 60-second periods with a one minute cooling time between grinds for a total of 5 minutes of grinding. Remove the dish and in a fume hood, open the lid and inspect the sample. It should be the consistency of flour. The consistency of the material is checked by

pinching some between fingers of a gloved hand and feeling for grit and by looking for any un-ground fibers. If grit is detected or if fibers are observed, additional grinding is needed.

NOTE: During the one-minute cooling time, the dish should be placed in a shallow ice water bath to facilitate cooling. Be sure the bath is shallow enough so that water does not get inside the dish.

10.5.2.6 If the sample reaches a flour-like consistency before all 5 one-minute grinds have been completed, then it might be beneficial to not perform all 5 grinds in order to avoid excessive heat and to avoid packing the sample onto the side of the grinder. If the analyst inspects the sample and it has a flour like consistency before all 5 grinds are completed, they can make the decision to stop after less than 5 grinds. A NCM should be written to document the deviation from the source method and the reasoning.

NOTE: If multiple 300 g increments are used for grinding and the sample is recombined, it has been shown in DU/TRL QC that the sample is non-homogenous. To re-homogenize the sample, place all volume in to a clean plastic bag, seal, and carefully shake the bag for 1-2 minutes until sample is homogenous. Lay out the sample back on the foil/parchment paper. This must be done on all samples regardless if this sample will be used for DU/TRL QC.

10.6 Aliquot the Samples

10.6.1 All aliquots should be taken using a square-ended disposable plastic spoon. This is done to ensure that finer sample material does not fall off of the sampling tool, as can happen if a spatula was used instead. This is particularly necessary when samples are not ground to a consistent grain size using the ring and puck.

10.6.2 **2 Gram Aliquot – Extraction Method "8330_P_2g"** – Remove the cap from a labeled 40 mL amber vial and place the vial on a balance and tare. Spread the entire sample out to a thickness no greater than 1 cm. Use a disposable plastic square-ended spoon to build a 2.0 g to 2.2 g aliquot by taking at least five small portions from random locations through the entire thickness of the sample. Record the exact sample weights on the benchsheet and cap the vial with a Teflon™ lined lid. Save the remaining soil for possible re-extraction. Create a LCS and a method blank by placing 2.0 g to 2.2 g of baked Ottawa sand in labeled vials. Record a nominal weight of 2 g in the initial volume field, then record the actual weight to the nearest 0.1 g in the notes column.

10.6.3 **10 Gram Aliquot – Extraction Method "8330_Sonc_10g"** – Remove the cap from a labeled 40 mL amber vial and place the vial on a balance and

tare. Spread the entire sample out to a thickness no greater than 1 cm. Use a disposable plastic square-ended spoon to build a 10 g to 11 g aliquot by taking at least thirty small portions from random locations through the entire thickness of the sample. Record the exact sample weights on the benchsheet and cap the vial with a Teflon™ lined lid. Save the remaining soil for possible re-extraction. Create a LCS and a method blank by placing 10 g to 11 g of baked Ottowa sand in labeled vials. Record a nominal weight of 2 g in the initial volume field, then record the actual weight to the nearest 0.1 g in the notes column. If the samples were ground create a grinding blank per Section 9.10, and take an aliquot from this composite. Aliquot the grinding LCS as you would a sample.

10.7 Add Surrogate, Spikes, and Solvent to the Samples

10.7.1 Refer to WI-DV-009 for the correct surrogate and spike standards to use and the correct volume.

10.7.2 The surrogate and spikes standards are kept in a freezer, but should be allowed to come to room temperature before use. Record the ID of the standard and pipette(s) used on the benchsheet.

10.7.3 The addition of spikes and surrogates to samples must be done only immediately after a second analyst has reviewed the batch. Reference work instruction WI-DV-009.

10.7.4 Only one batch should be surrogated at a time to ensure the correct standards are used.

10.7.5 Using a calibrated pipette, add the appropriate volume of the appropriate working surrogate standard to each sample and each QC sample.

10.7.6 Using a calibrated pipette, add the appropriate volume of the appropriate working spike standard to each LCS and MS/MSD.

NOTE: Do not add the spike standard to the grinding LCS. The grinding LCS is created using the material described in Section 7.5 and already contains the analytes of interest.

10.8 Add Extraction Solvent

10.8.1 2 Gram Extraction – Extraction Method “8330_P_2g”

10.8.1.1 Taking into account the volume of surrogate and spike standard added to each sample, bring the extract volume up to 10 mL with the soil extraction solvent described in Section 7.3. Use either a 10 mL Class A graduated cylinder or a bottle top pump that has been calibration checked.

Example: If 0.5 mL of surrogate standard was added to a sample, add exactly 9.5 mL of the soil extraction solvent.

Example: If 0.5 mL of surrogate standard and 0.5 mL of spike standard was added to a LCS, add exactly 9 mL of the soil extraction solvent.

10.8.1.2 Proceed to Section 10.9

10.8.2 10 Gram Extraction – Extraction Method "8330_Sonc_10g"

10.8.2.1 Taking into account the volume of surrogate and spike standard added to each sample, bring the extract volume up to 20 mL with the soil extraction solvent described in Section 7.3. Use either a 25 mL Class A graduated cylinder or a bottle top pump that has been calibration checked.

Example: If 1 mL of surrogate standard was added to a sample, add exactly 19 mL of the soil extraction solvent.

Example: If 1 mL of surrogate standard and 1 mL of spike standard was added to a LCS, add exactly 18 mL of the soil extraction solvent.

10.9 Extract the Samples

10.9.1 Cap vial with a Teflon-lined cap, vigorously hand shake the vial for one minute, or until all material is well mixed, and place it in a box. Place the box on the platform shaker so that the vials are lying on their side. Set the platform shaker at 150 rpm and allow the samples to be shaken for at least 18 hours. Record the start time on the benchsheet.

10.9.2 After the 18 hour extraction, remove the vials from the shaker table and record the stop time on the benchsheet.

10.9.3 If needed, centrifuge the vial at no more than 2200 rpm to help separate the solids from the extract. Remove approximately 10 mL of the supernatant solution. Filter the supernatant solution using a 0.2-μm PTFE syringe discarding the first mL into the waste. Filter the remaining supernatant into a labeled 8-mL amber vial.

10.9.4 Submit the extract for analysis to the appropriate analytical lab.

10.10 Maintenance

10.10.1 Approximately once a month, the cover on the Ring and Puck should be removed and any dirt should be cleaned up.

10.10.2 When excessive wear is noted, replace the hammers in the Mechanical Grinder.

10.10.3 Occasional lubrication of the Ring and Puck clamp is needed.

10.10.4 The o-rings in the Ring and Puck dishes should be replaced when worn.

10.10.5 Every 6 months the centrifuge should be lubricated and tightened.

10.11 Troubleshooting

The Ring and Puck dishes are all slightly different depths. Therefore the clamp that holds them to the grinder does not fit snuggly on all dishes without the use of a pad. It is important to have a snug fit to ensure the dish lid seals tightly to avoid sample loss.

11.0 Method Performance

11.1 Method Detection Limit (MDL)

A valid method detection limit (MDL) study for each analyte of interest must be performed prior to analyzing samples for the first time and verified annually thereafter. Separate soil MDL studies are performed for 8330A using 2 g and 8330B using 10 g of Ottawa sand. Separate soil MDL studies are performed for explosive method 8321A using 2 g of Ottowa sand and 8321A using 10 g of Ottowa sand. A MDL study for picric acid by method 8321A is performed using 10 g of Ottowa sand. A MDL study for explosives by 8321 LC/MS/MS is performed using 10 g of Ottowa sand. The procedure for determining detection limits is defined in Policy DV-QA-005P. Quarterly MDLV and LOQV studies are performed for the DoD program (QSM 4.2 and 5.0)

11.2 Limit of Quantitation Verification (LOQV)

The verification of the limit of quantitation (LOQ or LLOQ) is performed quarterly for work performed according to the DOD/DOE QSM 5.0 or for programs that specify the requirement. A blank matrix is spiked at 1-2 the laboratory RL and carried through the entire preparation and analytical procedures. Recoveries are assessed based on historical limits.

11.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

11.3.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

11.3.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

11.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

11.3.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

11.3.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

11.4 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

12.0 Pollution Control

Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

13.0 Waste Management

13.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

13.2 The following waste streams are produced when this method is carried out:

13.2.1 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

13.2.2 Flammable solvent waste – Waste Stream C

13.2.3 Solid sample waste - Waste Stream D

13.2.4 Waste soil sample vials - Waste Stream A

NOTE: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

14.0 References / Cross-References

14.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

14.1.1 Method 8330, Nitroaromatics and Nitramines by High Performance Liquid Chromatography, Revision 0, September 1994.

14.1.2 Method 8000B, Determinative Chromatographic Separations, Revision 2, December 1996.

14.1.3 Method 8330A, Nitroaromatics and Nitramines by High Performance Liquid Chromatography, Revision 1, January 1998.

14.1.4 Method 8330B, Nitroaromatics, Nitramines, and Nitrate Esters by High Performance Liquid Chromatography, Revision 2, October 2006.

14.2 DoD Environmental Data Quality Workgroup, Frequently Asked Questions (FAQs) Concerning the Implementation of EPA SW-846 Method 8330B, November, 2014.

15.0 Method Modifications:

15.1 Method 8330 prescribes the shelf life for standards as follows:

Standards	Concentration	Shelf Life
Stock standards	1,000,000 µg/L (1,000 ppm)	One year
Intermediate standards	2.5 to 1,000 µg/L	Thirty days
Working standards	1 to 500 µg/L	Daily

This SOP assigns a six-month shelf life to the working level standard based on TestAmerica's experience with these materials. The standards are stored in a freezer.

16.0 Attachments

Table 1. Analyte List
Table 2. Grinding LCS Bulk Material
Appendix 1 Flowchart for Drying Explosive Soils
Appendix 2 Flowchart for Grinding and Sieving Soils
Appendix 3 Instructions for Batching in TALS
Appendix 4 ISM Worksheet
Appendix 5 ISM Constant Weight Worksheet

17.0 Revision History

- Revision 9, dated 5 October 2017
  - Added wording to 2.1 to clarify that samples are dried to a constant weight.
  - Added 10.3.8 and 10.3.9 and removed 15.2 to include procedure for drying samples to a constant weight.
  - Added Appendix 5

- Revision 8, dated 2 December 2016
  - Added the comment requiring the documentation of equipment IDs to Section 6.1
  - Added Section 6.3 Computer Software and Hardware
  - Added Note to Section 9.5 requiring a LCSD when there is no volume for MS/MSD
  - Added Note to Section 10.0 regarding the rotation of sieves and equipment
  - Updated Section 10.5.2.4 to include the samples should be ground the same length

of time as the LCSSRM, renumbered the notes and included a sample grind time exception.
  - Updated Section 10.5.3.5 to include Note 1 from Section 10.5.3.6.
  - Renumbered Note in Section 10.5.3.6
  - Updated Section 10.7.2 to reflect standard SOP
  - Removed the Ball Mill Grinder reference from the entire SOP.
- Revision 7, dated 31 January 2016
  - Annual Technical Review
  - Deleted previous Section 4.3 no longer applied – vegetation and rocks are removed
  - Added Section 4.6 regarding tetryl decomposing with exposure to heat.
  - Added paragraph to Section 7 to contain reagent grade verbiage consistent with other SOPs
  - Section 8.1 – clarified the paragraph to be specifically about method 8330A
  - Added “and not frozen” to section 8.3
  - Revised Section 9.1 to have consistent verbiage and instructions as other SOPs
  - Added Note to Section 9.6 regarding DoD MS/MSD requirements
  - Changed duplicate to triplicate in Section 9.9
  - Clarified instruction to not clean the ring and puck dish after the blank in Section 10.5.3.3
  - Added the weight recording requirements to Sections 10.6.2 & 10.6.3
  - Added “and pipette(s)” to Section 10.7.2
  - Changed the centrifuge speed from 2500 rpm to 2200 rpm and 5mL to 10mL volume of supernatant solution to remove in Section 10.9.3
  - Modified/Rearranged Section 11 to be consistent with other SOPs
  - Removed previous Section 11.1 “Initial Demonstration of Capability”
  - Added current Sections 11.2 “Demonstrations of Capabilities” & 11.3 “Training Requirements”
  - Reformatted Section 14 and added Section 14.2 – reference to DoD Frequently Asked Questions for 8330B
  - Removed references to AFCEE and USACOE throughout document as these programs were incorporated into the DoD program.
  - Removed all 2010 and earlier revision histories
- Revision 6, dated 31 January 2015
  - Annual Technical Review
  - Reformatted SOP.
  - Revised Section 3.7 and Section 9.2 to state that a Ball Mill grinding batch is opened and closed the same day, while a Ring and Puck grinding batch can be open for up to 3 days.
  - Revised Section 5.2.2 to give information on the hazards of the Humbolt grinder.
  - Revised Section 9.9.1 to give more detail on how the Ring and Puck composite grinding blanks are created.
  - Revised Section 10.3.2 and Appendix 1 to state that any sample logged with an ISM method must have the entire sample container dried.
  - Revised Section 10.10 to include maintenance on the centrifuge.
- Revision 5, dated 27 January 2014
  - Annual Technical Review
  - Removed Section 1.2.3, DV-LC-0028 no longer performed.
  - Added detail about sieve size to Section 1.3.2.
  - Edited Section 6.1, subsection “Ball Mill” to allow for un-baked sand to be used in the cleaning of the ball mill stones and to allow the use of 1 pint cans. The section was

also revised to change the minimum time the stones have to be tumbled during the cleaning process from 3 hours to 2 hours. This was done based on analyst's experience.
  - Edited Section 6.1, subsection "Sieves" to state a brillo pad can be used on the sieves so long as the mesh is not damaged.
  - Updated Section 9.1 to reflect current practice, added a comment stating that this procedure meets DoD QSM 5.0 criteria unless otherwise stated.
  - Removed Acceptance Criteria and Corrective Action information to Section 9. This information can be found in the analytical SOPs.
  - Revised Section 9.6 to state that if there is no volume for a MS/MSD, a LCSD must be performed.
  - Added information to Section 9.9 for DoD acceptance criteria for triplicates.
  - Updated sections 10.1, 10.2 and 11.2 to reflect current practice
  - Added a NOTE in Sections 10.5.2.1 and 10.5.3.6 giving instructions on how to ensure the sample is homogenous after it has been split into separate grinding containers and then later re-combined.
  - Added Section 10.5.3.6 giving guidance on what to do if the sample reaches a flour-like consistency before all 5 grinds have been completed. This was done to avoid over-heating samples and packing the sample against the grinding dish wall.
  - Added Section 10.10 Maintenance and Section 10.11 Troubleshooting per DoD QSM 5.0.
  - Updated Appendix 2 and Appendix 3 to reflect the current method names used in LIMS.
  - Formatting changes throughout
- Revision 4, dated 30 October 2012
  - Annual Technical Review
  - Section 4.6 was added to document the adverse affect headspace in the ball mill can has on the grinding LCS.
  - Section 6.1 and Section 10 were revised to include the description of the Spacer Can in the Ball Mill apparatus.
  - Section 6.1 and Section 10 were revised to include the Mechanical Grinder used as an alternative to mortar and pestle.
  - Section 9.6 was revised to state that LCSDs are not required for DoD work.
  - Section 10 was revised to reference the Explosive Review Checklist in WI-DV-0009.
  - Section 10.3.2 was revised to instruct the analyst to eliminate as much headspace as possible during the Ball Mill grinding step.
  - Appendix 3 was revised to give more detail on the steps taken to ensure all pre-ground ISM aliquots are taken before the sample is ground. It was also revised to include the use of the Explosive Extraction Checklist in WI-DV-0009.
- Revision 3, dated 10 October 2011
  - The procedure was revised to have the extraction performed by shaker table instead of cooled sonication bath. This was done to increase lab capacity and to create a more rugged extraction.
  - Section 5 was revised to include the requirement that analysts wear cut-resistant gloves when tightening vial caps.
  - Section 7.2 was revised to include the lot approval process for acetonitrile.
  - Sections 7.5 and 9.3 were revised to mandate a 3 day expiration date on the Grinding LCS after it has been ground.
  - Section 9.8 and 9.9 were revised to require a duplicate and triplicate whenever method 8330B is performed, not just when samples are ground.

- Section 10.2.2 and 10.2.3 were revised to have the analyst use the prep method instead of the pre-prep method to determine sieve size. This is a simpler determination and matches the flow chart in Appendix 2.
- Section 10.7.3 was revised to change the speed of the centrifuge to prevent the breakage of the extract vials.

- Revision 2, dated 11 January 2011
  - Details about the surrogate and spike standards used in this procedure have been moved to SOP DV-OP-0020.
  - Revised Section 9 to state that duplicates and triplicates are required when ring and puck or ball mill grinding is performed.
  - Revised the procedure to include instructions and details for the laboratory's new LIMS.
  - Revised Section 4 to give more details on the grinding of samples.
  - The procedure was revised to state that samples should be ground on the ball mill for only 8 hours. At that time, the samples should be inspected and only ground longer if required.
  - Added detail in Section 10.1 about the electronic temperature monitoring device that records the temperature of the drying room.
  - Revised the flowcharts to be flowcharts only and not worksheets. All data is now recorded in TALs benchsheets.
  - Added instructions in Appendix 3 on how to batch samples in TALSs
  - Added the option to use an automatic sieve shaker.

Earlier revision histories have been archived and are available upon request.

Table 1. Analyte List

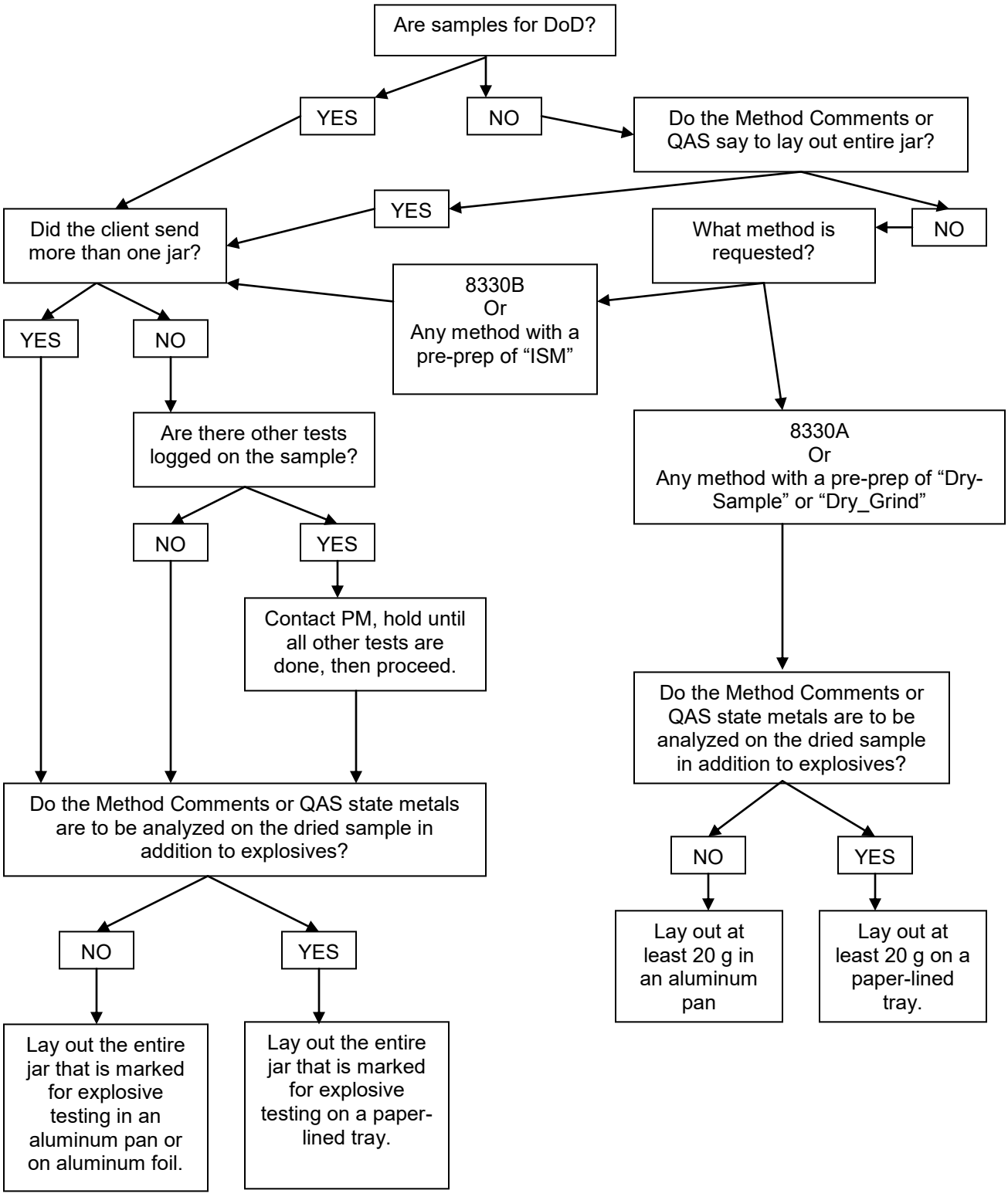
Compound	CAS #	Symbol
Octahydro-1,3,5,7-tetranitro-1,3,5,7,-tetrazocine	2691-41-0	HMX
Hexahydro-1,3,5-trinitro-1,3,5-triazine	121-82-4	RDX
1,3,5-Trinitrobenzene	99-35-4	1,3,5-TNB
1,3-Dinitrobenzene	99-65-0	1,3-DNB
Methyl-2,4,6-trinitrophenyl nitramine	479-45-8	Tetryl
Nitrobenzene	98-95-3	NB
2,4,6-Trinitrobenzene	118-96-7	2,4,6-TNT
4-Amino-2,6-dinitrotoluene	19406-51-0	4-Am-DNT
2-Amino-4,6-dinitrotoluene	35572-78-2	2-Am-DNT
2,6-Dinitrotoluene	606-20-2	2,6-DNT
2,4-Dinitrotoluene	121-14-2	2,4-DNT
2-Nitrotoluene	88-72-2	2-NT
4-Nitrotoluene	99-99-0	4-NT
3-Nitrotoluene	99-08-1	3-NT
Nitroglycerin	55-63-0	NG
PETN	78-11-5	PETN
2,4-Diamino-6-nitrotoluene**	6629-29-4	--
2,6-Diamino-4-nitrotoluene**	59229-75-3	--
Picric Acid	88-89-1	PA
1-Nitroso-3,5-dinitro-hexahydro-1,3,5-triazine**	5755-27-1	MNX
3,5-Dinitroaniline**	618-87-1	3,5-DNA
1,2-Dinitrobenzene (8330 surrogate)	528-29-0	1,2-DNB
Nitrobenzene-d5 (8321 surrogate)	--	NB-d5

** Compounds are only analyzed and spiked upon request.

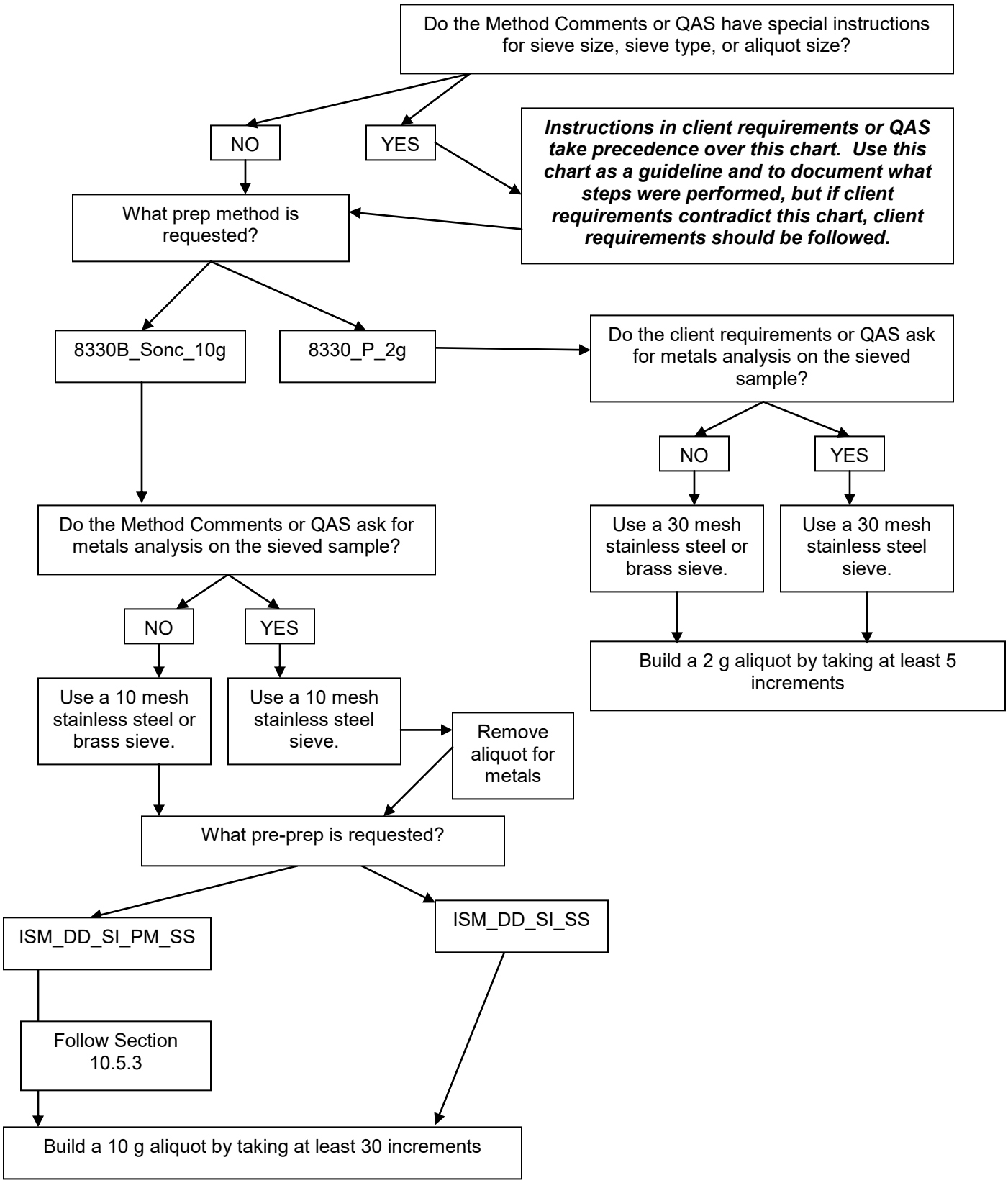
Table 2. Grinding LCS Bulk Material

Compound	Concentration (µg/Kg)
4-Amino-2,6-dinitrotoluene	600
2-Amino-4,6-dinitrotoluene	600
1,3-Dinitrobenzene	600
2,4-Dinitrotoluene	600
2,6-Dinitrotoluene	600
HMX (Octahydro-1,3,5,7-TNTC)	600
1,3,5-Trinitrobenzene	600
Nitrobenzene	600
2-Nitrotoluene	600
3-Nitrotoluene	600
4-Nitrotoluene	600
RDX (Hexahydro-1,3,5-TNTriaz)	600
Tetryl (Methyl-2,4,6-TNPN)	600
Nitroglycerin (Trinitroglycerin)	600
Pentaerythritol tetranitrate (PETN)	600
2,4,6-Trinitrotoluene	600

Appendix 1 – Flowchart and Worksheet for Drying Explosive Soils



Appendix 2 – Flowchart and Worksheet for Grinding and Sieving Explosive Soils



Appendix 3

How to Batch:

ISM_DD_SI_PM_SS (Dry, Disaggregate, Sieve, Ring & Puck, Subsample)
ISM_DD_SI_SS (Dry, Disaggreage, Sieve, Subsample)
Dry_Sample (Dry, Sieve, 2g prep)
Dry_Grind (Dry, Sieve, 2g Prep)

Overview
These five pre-prep methods can be logged in for not just for samples for explosives by 8330A or 8330B and 8321A or 8321B, but also for samples for metals analysis, or perchlorate, or any other method where the client is asking the lab to dry, sieve, and possibly grind the sample before extraction or digestion.

If one sample is logged in for 8330B and 6010B and 6020B and 7471A and all of these methods have the pre-prep of ISM_DD_SI_SS, the sample will show up on the backlog 4 times, (once for each analytical method). This would happen if the client wants us to dry, sieve, and perform ISM for each of these methods.

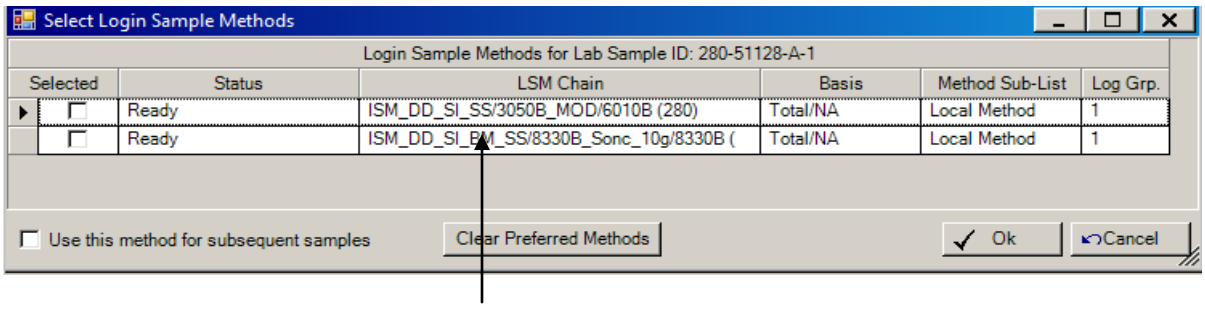
If one sample is logged in for 8330B with a pre-prep of ISM_DD_SI_PM_SS and the same sample is logged in for method 6010B with a pre-prep of ISM_DD_SI_SS, that means that the client wants us to dry, sieve the sample, perform ISM for method 6010B, then ring and puck and perform ISM for 8330B.

We will use a different status to indicate where the samples are.

- A status of “Batched” means the samples have been laid out to dry.
- A status of “Scheduled” on ISM_DD_SI_PM_SS means the samples have been laid out to dry, but possibly need ISM performed before grinding.
- A status of “Partial” means the samples have been sieved.
- A status of “2nd Level Review” on methods ISM_DD_SI_SS, Dry_Sample, or Dry_Grind means that the aliquots have been taken and someone has checked your work.
- A status of “2nd Level Review” on ISM_DD_SI_PM_SS means samples have completed the grinding.

Steps for Samples logged for <u>both</u> ISM_DD_SI_SS <u>and</u> Ring & Puck

1. Run the Dry/Sieve/Grind/ISM backlog. This backlog will only have samples that are logged in for these five pre-preps. This backlog is sorted by sample ID so that if a sample is logged in for ISM_DD_SI_SS for metals and ISM_DD_SI_PM_SS for explosives, you can easily see that the sample needs both preps.
2. Batch the samples under the ISM_DD_SI_SS method.
   NOTE: Do not put samples in the same batch that require different sieve sizes.
3. Scan your samples into the batch. A window will appear called “Select Login Sample Methods”.



4. Select only the methods that have ISM_DD_SI_SS as the pre-prep.
    a. You can only batch ISM samples under a ISM_DD_SI_SS batch.
    b. You can only batch ring and puck samples under a ISM_DD_SI_PM_SS batch.
    c. You can batch “Dry_Grind” and “Dry Sample” samples under the same batch.
5. Save the batch and print the benchsheet.
6. Print labels. Print one label for each method logged on each sample. Write on the label the analytical method.
7. Lay out the samples to dry. Place all of the labels on the sample tray for the sample. Also label the tray “Grinding Needed” if the sample is logged for Ring and Puck.
8. Set the Ring and Puck Methods to “Scheduled” in the backlog. Do not batch them at this time.
9. Sieve the samples and take the required ISM aliquots. Whenever possible, aliquot the samples directly in the digestion cup for metals, or microwave tube or beaker for organics and record the aliquot masses in the ISM worksheet in Appendix 4. Place the ISM aliquots on the tray with the sample so a 2nd analyst can perform a label check. Document these steps on the TALS batch sheet
10. The Notes field in the Worksheets tab can be used to document if there was rocks or vegetation that did not go thru the sieve. Write NCMs for any samples that contained rocks or vegetation that was removed from the sample.
11. In the Worksheet tab, you can record the weight of the sample before and after drying and the weight of the sample that went through the sieve and the weight of the sample that did not go through the sieve. These measurements are not normally required, so they only need to be performed if client requested.
12. Have a 2nd analyst review the ISM_DD_SI_SS batch to ensure all required ISM aliquots have been performed. Take the ISM_DD_SI_SS batch to 2nd level review.
13. Now the samples are ready to be ground by Ring and Puck.
14. As the samples are ground, add them to the ISM_DD_SI_PM_SS batch.
15. As you add samples to the grinding batch, watch the LSM window to ensure that all ISM_DD_SI_SS methods are at 2nd level review. If there are ISM_DD_SI_SS methods that are not at 2nd level review, perform ISM on the sample for the requested methods before grinding the sample.
16. Take the samples to 2nd level review.
17. Return all empty containers to the walk-in refrigerator using ICOC. Any left-over dried and ground material is stored in the walk-in refrigerator on the same shelf as the original client containers.

Steps for Samples logged for Ring & Puck ONLY. No ISM_DD_SI_SS methods logged.

1. Run the Dry/Sieve/Grind/ISM backlog. This backlog will only have samples that are logged in for these five pre-preps. This backlog is sorted by sample ID so that if a sample is logged in for ISM_DD_SI_SS for metals and ISM_DD_SI_PM_SS for explosives, you can easily see that the sample needs both preps.
2. Pull the samples from the walk-in cooler and take custody of the samples. Take note of what shelf the sample came from.
3. Batch the samples and print out labels. Then remove the samples from the batch to place them back on the backlog.
4. Lay the samples out on parchment or foil. Label each tray with the sample ID and the grinding method (Ring & Puck)
5. Document the date and time the samples were laid out to dry. Document if the samples were laid out on parchment or foil. Document that a label check was performed.
6. Set the samples to Scheduled in the backlog to show that they are laid out to dry.

7. Once the samples are dry enough to sieve, sieve the samples and document what sieve size on the Explosive Review Checklist.
8. Open the batch with a grinding LCS. As the samples are ground, add them back to the original ISM_DD_SI_PM_SS batch. There can only be 20 field samples in each batch,. Ring and puck batches can be open for up to 3 days.
9. As you add samples to the grinding batch, watch the LSM window to ensure that the samples do NOT require any non-ground aliquots.
10. Take the samples to 2nd level review.
11. Return all empty containers to the walk-in refrigerator using ICOC. Any left-over dried and ground material is stored in the walk-in refrigerator on the same shelf as the original client containers.

Steps for Samples logged for <u>only</u> ISM_DD_SI_SS, Dry_Grind, or Dry_Sample. No grinding methods logged.

1. Run the Dry/Sieve/Grind/ISM backlog. This backlog will only have samples that are logged in for these five pre-preps. This backlog is sorted by sample ID so that if a sample is logged in for ISM_DD_SI_SS for metals and ISM_DD_SI_PM_SS for explosives, you can easily see that the sample needs both preps.
2. Batch the samples under the pre-prep method logged. Do not put samples in the same batch that are logged for different pre-prep methods. Do not put samples in the same batch that require different sieve sizes.
3. Scan your samples into the batch. If your samples are logged in for more than one of these three methods, a window will appear called “Select Login Sample Methods”.
4. Select only the methods that have ISM_DD_SI_SS as the pre-prep.
    a. You can only batch ISM samples under a ISM_DD_SI_SS batch.
    b. You can batch “Dry_Grind” and “Dry Sample” samples under the same batch.

    If the LSM window shows methods with pre-preps of ISM_DD_SI_PM_SS or ISM_DD_SI_BM_SS, then stop and follow the instructions above under the header “Steps for Samples logged for <u>both</u> ISM_DD_SI_PM_SS and Ring & Puck.
5. Save the batch and print the benchsheet.
6. Print labels. Print one label for each method logged on each sample. Write on the label the analytical method.
7. Lay out the samples to dry. Place all of the labels on the sample tray for the sample.
8. Sieve the samples and take the required aliquots. Whenever possible, aliquot the samples directly in the digestion cup for metals, or microwave tube or beaker for organics and record the aliquot masses in the ISM worksheet in Appendix 4. Place the aliquots on the tray with the sample so a 2nd analyst can perform a label check.
9. The Notes field in the Worksheets tab can be used to document if there was rocks or vegetation that did not go thru the sieve. Write NCMs for any samples that contained rocks or vegetation that was removed from the sample.
10. In the Worksheet tab, you can record the weight of the sample before and after drying and the weight of the sample that went through the sieve and the weight of the sample that did not go through the sieve. These measurements are not normally required, so they only need to be performed if client requested.
11. Have a 2nd analyst review the batch to ensure all required aliquots have been performed. Take the batch to 2nd level review.
12. Return all empty containers to the walk-in refrigerator using ICOC. Any left-over dried and ground material is stored in the walk-in refrigerator on the same shelf as the original client containers.

Appendix 4

ISM Worksheet

G:/QA/Edit/FORMS/Organic Prep Forms/MASTER ISM Spreadsheet_Rev1

ISM BATCH:

Use this spreadsheet to document alquot weights when aliquotting into digestion or extraction vessels. If aliquoting into a temporary vessel, no need to document the exact weight because the sample aliquot will be transferred and weighed at the time of analysis.

Login	Sample	Method --->											
			(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
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Appendix 5

ISM Constant Weight Worksheet

Located: \\tafs\Lab2\Denver\Admin\QA\Edit\FORMS\Organic Prep Forms

DV-F-0070-10-03-2017-Rev 0

ISM Batch Number:

Use this spreadsheet to document drying samples to a constant weight.

			Analyst Initial Date/time 1	Analyst Initial Date/time 2	Dry?		Analyst Initial Date/time 3	Dry?	
Login	Sample	Tare weight (weight of weighing vessel)	Gross weight (g) (weight of aliquot and weighing vessel)	Gross weight (g) (weight of aliquot and weighing vessel)	Change in weights 1, 2	Proceed with ISM?	Gross weight (g) (weight of aliquot and weighing vessel)	Change in weights 2, 3	Proceed with ISM?
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